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ACTIVE CLEANING TECHNIQUE FOR REMOVING CONTAMINATION FROM OPTICAL SURFACES IN SPACE

Quarterly Progress Report No. 1

February 1 to May 1, 1971

by

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1.0 SUMMARY

This progress report discusses work accomplished during the first three-month period of a program aimed at developing an active cleaning technique (ACT) for removing contaminants from optical surfaces in space. The first year of this effort will concentrate on proving the feasibility of using a reactive gas plasma for removing contaminants from surfaces in vacuum. In addition, an operational parameter study will be conducted on a space-use cleaning device, and a preliminary design will be prepared of a laboratory-model ACT which could later be flown in space.

The work accomplished during this reporting period includes design and preparation of test facilities, preparation of test specimens, experiments aimed at optimizing the design of the plasma cleaning device, and experiments aimed at establishing the design of a suitable device for depositing liquid contaminants on specimens in vacuum.

2.0 INTRODUCTION

The need for developing an in-situ or active cleaning technique (ACT) for use in both space and vacuum chambers has recently become apparent. Manned spacecraft have experienced numerous contamination problems including deposition of volatile organic compounds onto windows, and light scattering from particulate contaminants surrounding the spacecraft. Sources of this contamination include outgassing of organic compounds, waste and water dumps, rocket plumes, and leakage from the life support system. It is believed that contaminant film deposition has also occurred on unmanned spacecraft surfaces. Data from a reflectometer experiment on the ATS-3 spacecraft (Reference 1) has indicated rather severe degradation on reflective surfaces, which may be the result of contaminant film deposition. Also, gratings in the extreme UV solar scanning experiment on OSO-V have degraded -- presumably the result of contaminant film deposition. Contamination on an unmanned spacecraft has been verified with quartz-crystal thin film monitors on OGO-6 (Reference 2). A recent review of the spacecraft contamination problem has been published in Reference 3.

Contamination can also occur during spacecraft testing in high vacuum chambers. A recent example of this was the extreme-UV solar spectroheliometer experiment for the Apollo Telescope Mount (ATM) vehicle. A film of back-streamed diffusion pump oil was apparently deposited on surfaces during thermal/vacuum testing (Reference 4). Another example of contaminant film deposition during environmental testing is discussed in Reference 5. In those experiments it was shown that an extremely stable organic film could be deposited onto telescope mirror surfaces during irradiation with low energy protons, in a relatively clean vacuum environment.

Based on existing knowledge, contamination problems anticipated for future spacecraft include: (1) deposition of non-volatile substances onto optical components, sensing elements, and temperature control surfaces; (2) particulate and gaseous contamination near the spacecraft (resulting in light scattering and absorption); and (3) chemical contamination which can interfere with upper atmosphere studies, analyses of interplanetary or planetary matter, and material processing experiments. It is anticipated that contamination effects can be reduced by changes in design, materials, operating procedures, and possibly control techniques. The use of more sensitive surfaces and longer term missions will, however, offset these improvements. Thus, the need exists for developing an ACT for space use.

The specific approach being investigated in this program involves exposing surfaces to a plasma containing atomic oxygen or combinations of other reactive gases. Experiments in Reference 5 have shown that this cleaning technique is very effective for removing contaminant films from optical surfaces in vacuum. Although detailed mechanisms of this process have not yet been studied, it is believed that oxidizable organic contaminant films are converted to volatile products such as CO_2 and H_2O which subsequently evaporate in vacuum. Recognizing that some contaminants will not be oxidizable into volatile compounds, cleaning by rf-sputtering will be evaluated on these materials.

In this research program, four different types of contaminant films will be applied to various types of mirrors, thermal control surfaces, gratings, and optical windows. The contaminants to be deposited will be derived from a typical hydrocarbon monomer (Styrene, $C_6H_5CH{:}CH_2$), a typical silicone monomer (Methyl trimethoxysilane, $CH_3Si(OCH_3)_3$, ethylene glycol, and urine. Coatings on mirrors will include Pt, Au, and $1/2\,\lambda$ and $3/4\,\lambda$ MgF_2 (at 121.6 nm) over Al. Coatings on gratings will include Pt and $1/2\,\lambda$ MgF_2 (at 121.6 nm) over Al. Thermal control surfaces will include treated zinc

oxide in methyl silicone (S-13G), zinc oxide in potassium silicate (Z-93), and silver-coated FEP Teflon.

3.0 PROGRAM PLAN

Since this is the first quarterly progress report, a brief description of the program plan is provided in this section.

The overall program plan is shown schematically in Figure 1. A reactive plasma cleaning technique is being investigated as discussed in Section 2.0. Following preparation of facilities and specimens, exploratory experiments will be conducted on one type of specimen using one type of contaminant. The purpose of these experiments will be to obtain data on the effects of varying contaminant deposition parameters, cleaning process parameters, and the need for in-situ reflectance measurements on certain specimens. This data will be useful for planning the remaining tests on other specimens.

If results of exploratory tests indicate success with the selected cleaning technique, comprehensive experiments will then be performed on other types of specimens and contaminants. In the event that certain types of contaminants cannot be removed by reactive plasma cleaning, a supplemental approach of rf-sputtering will then be evaluated as shown in Figure 1.

The experimental portion of the program will be followed by a spacecraft operational parameter study and a flight experiment preliminary design study. The objective of the former will be to define requirements and constraints for operating the proposed cleaning device in space.

Schematics showing additional details of both the exploratory and comprehensive test plans are shown in Figures 2 and 3, respectively. The exploratory tests (Figure 2) will be conducted on a mirror coated with

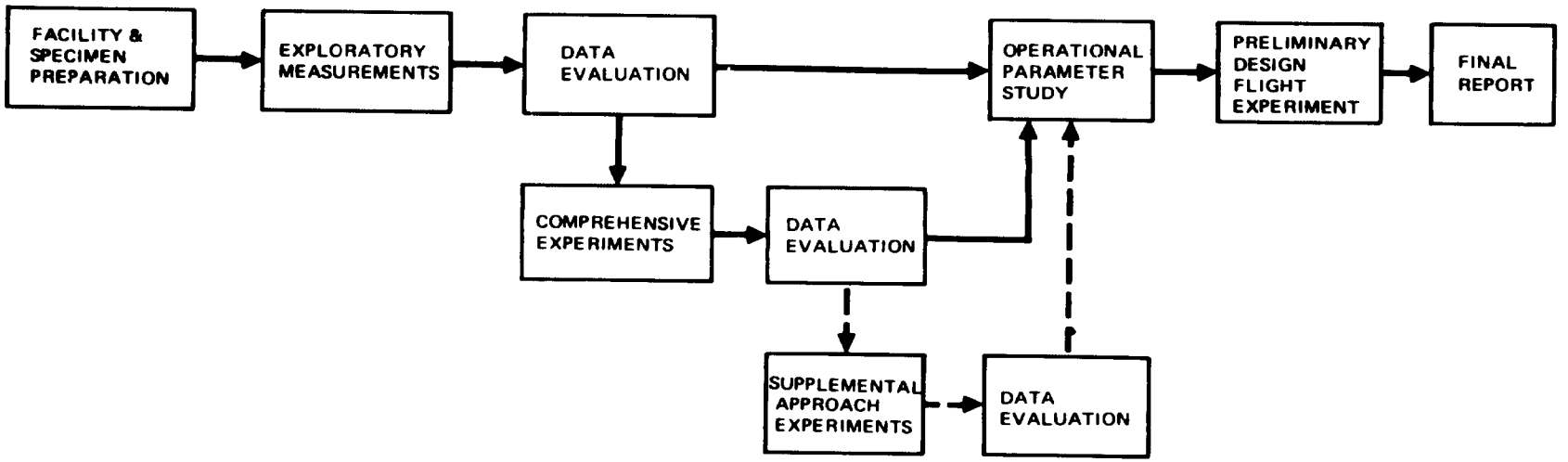
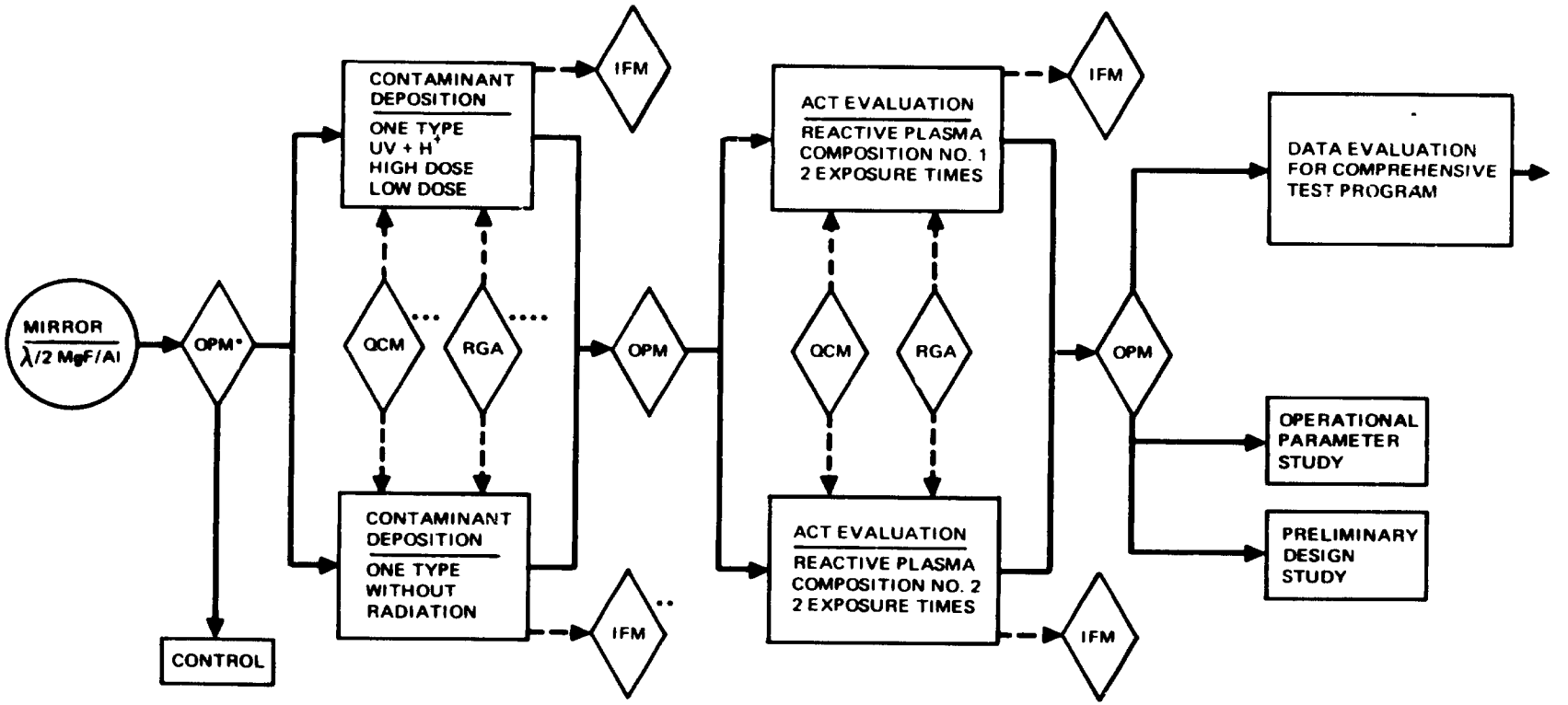


Figure 1: OVERALL PROGRAM PLAN



*OPTICAL PROPERTY MEASUREMENTS
**INTERFEROMETER MEASUREMENTS ON CALIBRATION MIRROR SPECIMEN
***QUARTZ CRYSTAL MONITOR
****RISIDUAL GAS ANALYSIS

Figure 2: SCHEMATIC OF EXPLORATORY TEST PLAN

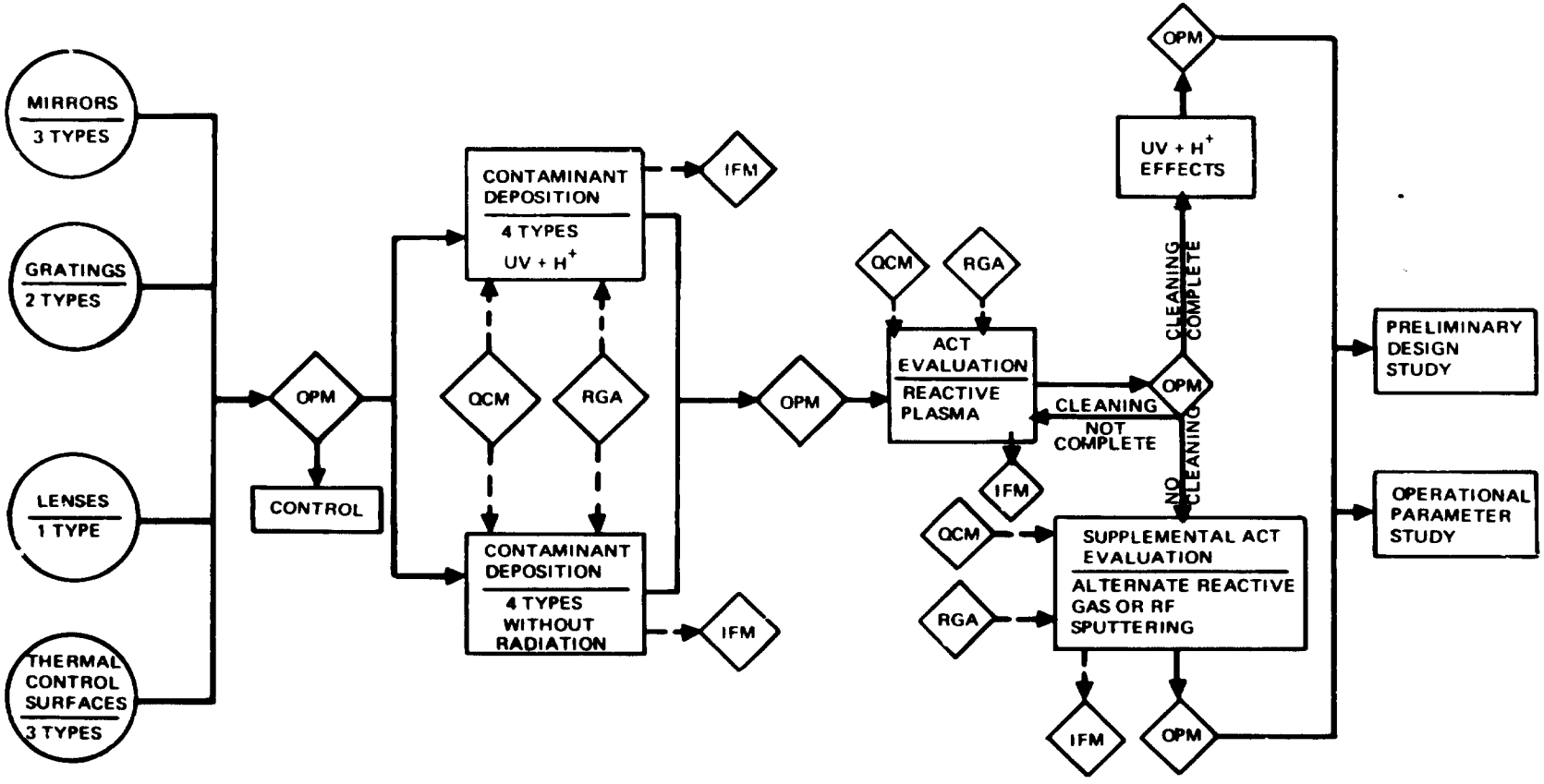


Figure 3: SCHEMATIC OF COMPREHENSIVE TEST PLAN

$\lambda/2$ MgF_2 on aluminum. Optical property measurements (OPM) will be made before and after contamination, and after cleaning. A control specimen from preliminary OPM will be saved for purposes of instrument calibration and to show whether specimens change with time in the laboratory environment. The need for performing in-situ reflectance measurements will be evaluated during exploratory tests. A hydrocarbon contaminant film will be applied at both a low and high radiation dose and without radiation to determine the effect of this parameter on cleanability. A residual gas analyzer (RGA) and quartz crystal monitor (QCM) will be used to monitor the vacuum chamber gas composition and film thickness, respectively, during deposition. An interferometer reference mirror (IFM) will be included in each run for absolute thickness measurements. The coated specimens will then be exposed to a reactive gas plasma at different plasma conditions and exposure times to determine the effect of these variables.

The comprehensive tests (Figure 3) will evaluate all types of specimens. This will include three types of mirrors, two types of gratings, one type of lens, and three types of thermal control surfaces. The specimens will be coated with the four types of contaminants outlined in Section 2.0. If the plasma successfully removes the contaminant film, specimens will be exposed to a UV + proton radiation environment to determine whether contamination and subsequent cleaning affects the radiation resistance. In the event that only partial cleaning occurred, a specimen would be re-exposed to the ACT. Specimens which are only partially cleaned or not cleaned at all by the oxygen plasma ACT will be subjected to a supplemental ACT. An alternate reactive plasma gas (such as H_2) would be tried first, and finally rf sputtering would be attempted using an inert gas such as argon.

4.0 TEST SPECIMENS

4.1 Mirrors

The 5.08 x 5.08 cm mirror test specimens were prepared from Pittsburgh Plate Glass; ground water-white glass. They were polished to within 10 fringes of sodium D wavelength light to provide a suitable optical surface for the ultraviolet reflectometer, and to reduce scattered light. Four different types of reflective surfaces were applied by vacuum deposition: MgF_2 over aluminum ($\lambda/2$ at 121.6 nm); MgF_2 over aluminum ($3\lambda/4$ at 121.6 nm); platinum (about 10 nm thick); and gold (about 500 nm thick). A flash NiCr adherence promoting coating was applied under both gold and platinum films. QCMs and IFMs were coated along with test mirrors.

4.2 Gratings

One-meter focal length gratings have been ordered from Diffraction Products, Inc. Four will be platinum coated and four will have magnesium fluoride over-coated aluminum surfaces. The specifications for these gratings are given in the following table.

Size	Focal Length	Blaze	Lines/mm	Surface
63.5 mm dia. x 12.5 mm thick	1 meter	60 nm	1200	Pt
Same	1 meter	150 nm	1200	Al/MgF_2 ($\lambda/2$ at 121.6 nm)

4.3 Thermal Control Surfaces

Three different types of thermal control surfaces have been furnished by the Government: treated zinc oxide in methyl silicone (S-13G): zinc oxide in potassium silicate (Z-93): and silver on FEP teflon. All three types of thermal control surfaces were bonded to aluminum substrates. Specimens are about 2.38 cm (15/16 in.) in diameter.

5.0 EXPERIMENTAL APPARATUS

5.1 Contaminant Film Deposition/Cleaning Facility

A schematic of the facility to be used for contaminant film deposition and cleaning experiments is shown in Figure 4. The multipurpose test chamber will employ subsystems including the plasma generator, contaminant vapor sources, a proton accelerator, an ultraviolet light source, and a residual gas analyzer.

The plasma generator consists of a source(s) of gas, suitable flow-measuring-and-controlling plumbing, three 4 mm ID quartz tubes, an rf power supply, and a capacitive antenna on the quartz tubes. The gas passes through the field, is dissociated, and flows through the quartz tubes into the test chamber. This arrangement will allow a lower pressure to exist in the test chamber than will exist in the plasma generator. The multiple tubes will distribute the reactive plasma gas over the test specimen holding plate.

Since the quartz tubes enter the chamber through an O-ring seal, the distance between them and the test specimens can be readily changed. Also, the size and number of tubes can be easily changed. This feature of the design was considered important because inadequate theory and empirical data exists at this time to confidently design the quartz tubes. Several experiments were run, as described later in Section 6.1, to obtain sufficient empirical data to choose the number and size of quartz tubes for this facility; however, it is likely that changes will have to be made during early experiments.

The contaminant vapor source, in the case of silane and hydrocarbon contaminants, will be a heated flask from which the monomer gases will

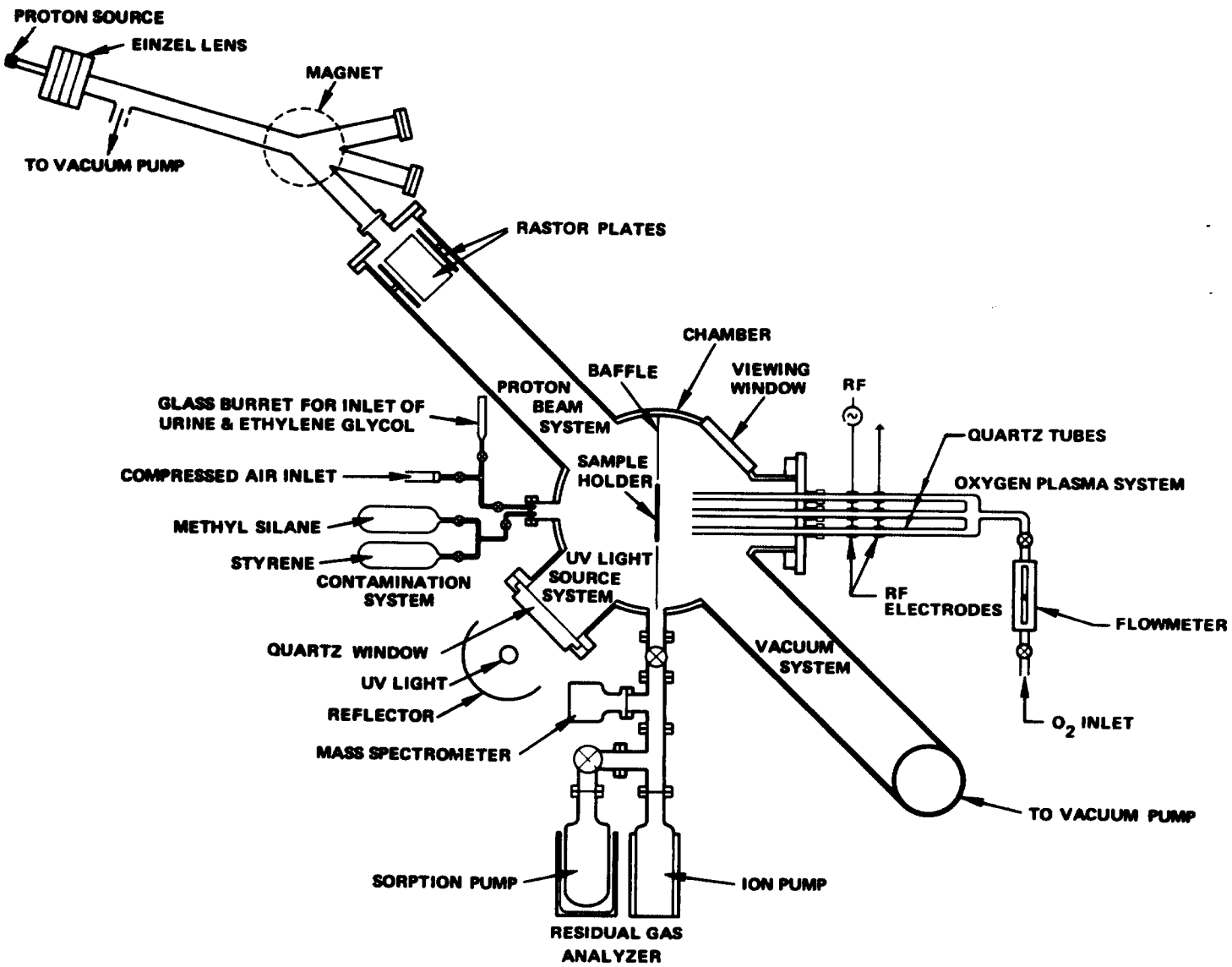


Figure 4: LOW TEMPERATURE OXYGEN PLASMA TEST FACILITY

be extracted. In the case of ethylene glycol, a spray nozzle will be installed in the chamber for spraying liquids in aerosol form onto the specimens. At the present time it is planned to spray urine aerosol onto the specimens in air, rather than in the chamber, because of the possible corrosive side effects on other components.

Proton and ultraviolet radiation will enter the test chamber through two different ports as shown in Figure 4, and impinge on the specimen surface. Low energy protons (1-10 keV) will be generated with an rf-discharge source utilizing electrostatic lenses for accelerating and focusing the particles. An electromagnet will be used for mass analysis, and a set of four charged plates will be used for rastering the proton beam over the entire test specimen holder area. A moveable Faraday cup will be used in the test chamber for determining the charged particle flux and uniformity across the test specimen array. Fluxes in the order of 10^{12} to 10^{13} protons/cm^2-sec will probably be used. Ultraviolet radiation will be produced with a General Electric UA-3, low-pressure mercury discharge lamp.

The residual gas analyzer (RGA) subsystem will utilize a Varian EAI Quad 150A quadrapole mass spectrometer as shown in Figure 4. The RGA subsystem will be evacuated with sorption and ion pumps and will be bakeable to remove residual contamination resulting from tests. A high-vacuum leak valve will separate the RGA from the main chamber. This will allow the mass spectrometer to operate at a much lower pressure (10^{-6} to 10^{-10} torr) than the main chamber, and thus increase sensitivity and minimize contamination.

A schematic showing the arrangement for supporting test specimens in the vacuum chamber is given in Figure 5. Three general types of specimens will be included in each contamination or cleaning run: (1) an optical test specimen (mirror, grating, transmitting optical element, or thermal

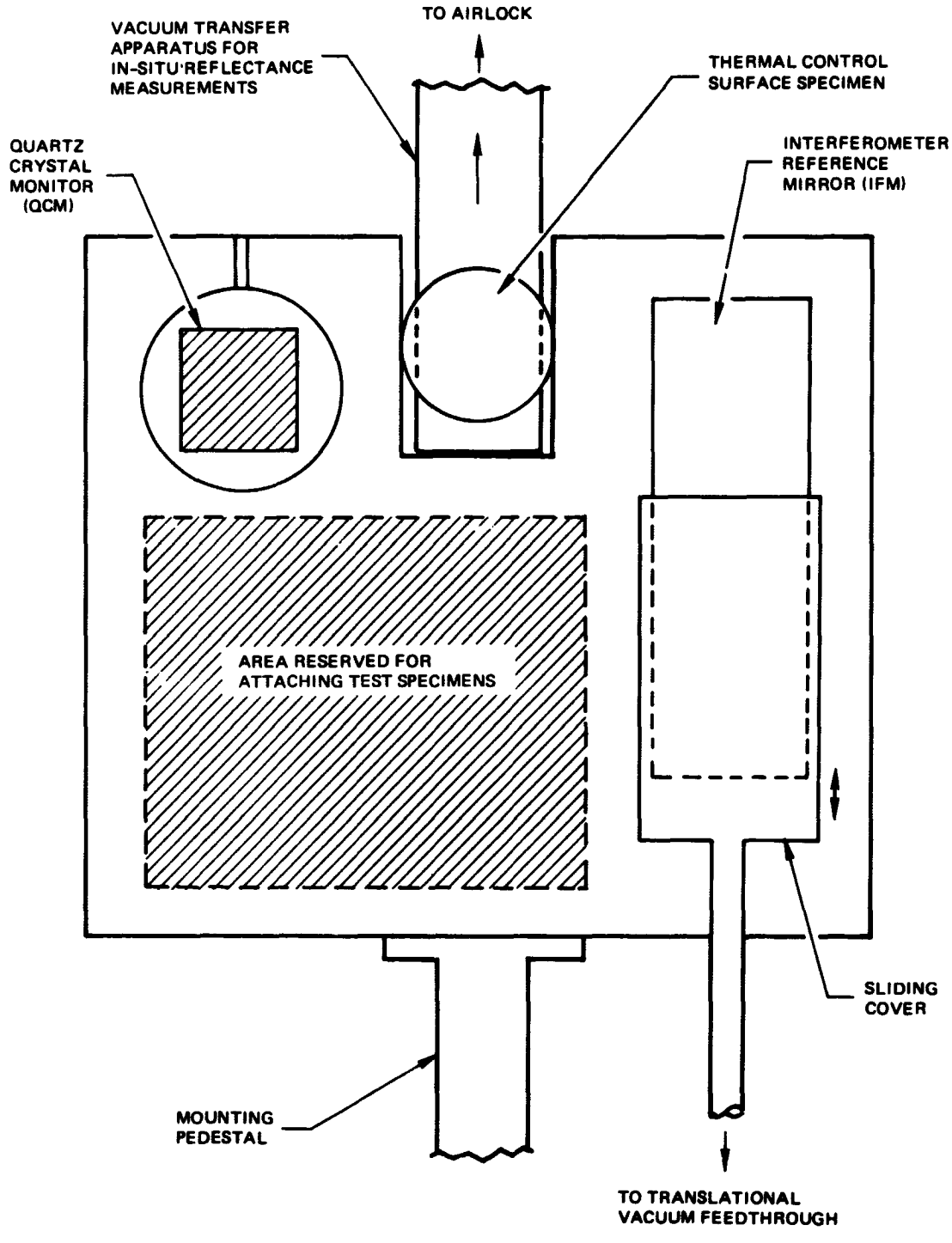


Figure 5 : SCHEMATIC OF TEST SPECIMEN HOLDING PLATE

control surface); (2) a QCM; and (3) an IFM. As shown in the figure, the IFM and QCM will be clamped to a temperature controlled plate which is projected into the cylindrical chamber from a rotatable vacuum mounting flange. The optical test specimen (in the case of mirrors and thermal control surfaces) can be either inserted into the chamber through a different port using a portable vacuum transfer apparatus, or can be mounted on the plate in the area shown.

The test specimen holding plate is electrically isolated from the chamber. This feature was incorporated to allow experiments to be conducted on the effects of electrical potential on contaminant film deposition. A study of this type is beyond the scope of the present program; however, it could easily be incorporated at a later time.

5.2 Film Thickness Measurements

Contaminant film thicknesses will be measured with QCMs and interferometer reference mirrors (IFMs). The QCMs being used are manufactured by Sloan Instruments, Inc. and are 5 MH_z AT-cut crystals. Because the QCM is sensitive to changes in film mass, the film thickness change can only be determined if the crystal has been directly calibrated or if the film density is accurately known. Experience has shown that a direct calibration of the QCM by multiple beam interferometry is the most reliable means of performing the required measurements. Calibration will be accomplished by measuring contaminant film thickness on IFMs, and correlating this thickness with QCM frequency change. During contaminant film deposition, a narrow strip on the surface of the IFM will be protected and thus will not get coated. Subsequently, the entire surface will be overcoated with vacuum deposited aluminum and examined with an interferometer. The thickness of the polymer film will be determined by measuring the fringe shift between the area coated with the contaminant and the protected area.

5.3 Ultraviolet Reflectance

Ultraviolet reflectance measurements on mirrors will be made with the facility shown in Figure 6. The system employs a McPherson Model 225 monochromator and a Boeing-fabricated specular reflectometer. The wavelength range capability with existing light source and optics is from about 95 nm to 500 nm. A specimen size of about 5.08 x 5.08 cm is required for this facility. Details of the ultraviolet reflectometer have been described in the literature (Reference 5) and thus will not be discussed here.

5.4 Solar Wavelength Reflectance

A schematic of the facility to be used for performing reflectance measurements on thermal control surfaces in vacuum is shown in Figure 7. Reflectance measurements are performed by locating the test specimen at the center of an integrating sphere (a Gier-Dunkle sphere arrangement). The spectral region from 0.20 to 2.5 microns can be scanned with the instrument. Typical pressure during reflectance measurements is in the order of 10^{-6} torr. The vacuum transfer apparatus, described earlier, is shown on the top of the integrating sphere.

5.5 Grating Efficiency

Grating efficiency (reflectance) measurements will be made using the apparatus shown in schematic form in Figure 8. Measurements will be made to determine the efficiency of reflectance in first order, where this efficiency is defined as the ratio of the intensity of radiation reflected in first order to the intensity of radiation of the same wavelength incident upon the grating. In operation, monochromatic radiation throughout the wavelength range 100-500 nm is obtained using a pulsed spark discharge, a high pressure mercury lamp, and a Seya vacuum monochromator. The dispersed radiation entering the reflectometer falls either on the photomultiplier (sensitized with sodium salicylate for the vacuum UV region) at position PM_1, or on the grating (g) being studied. Radiation reflected

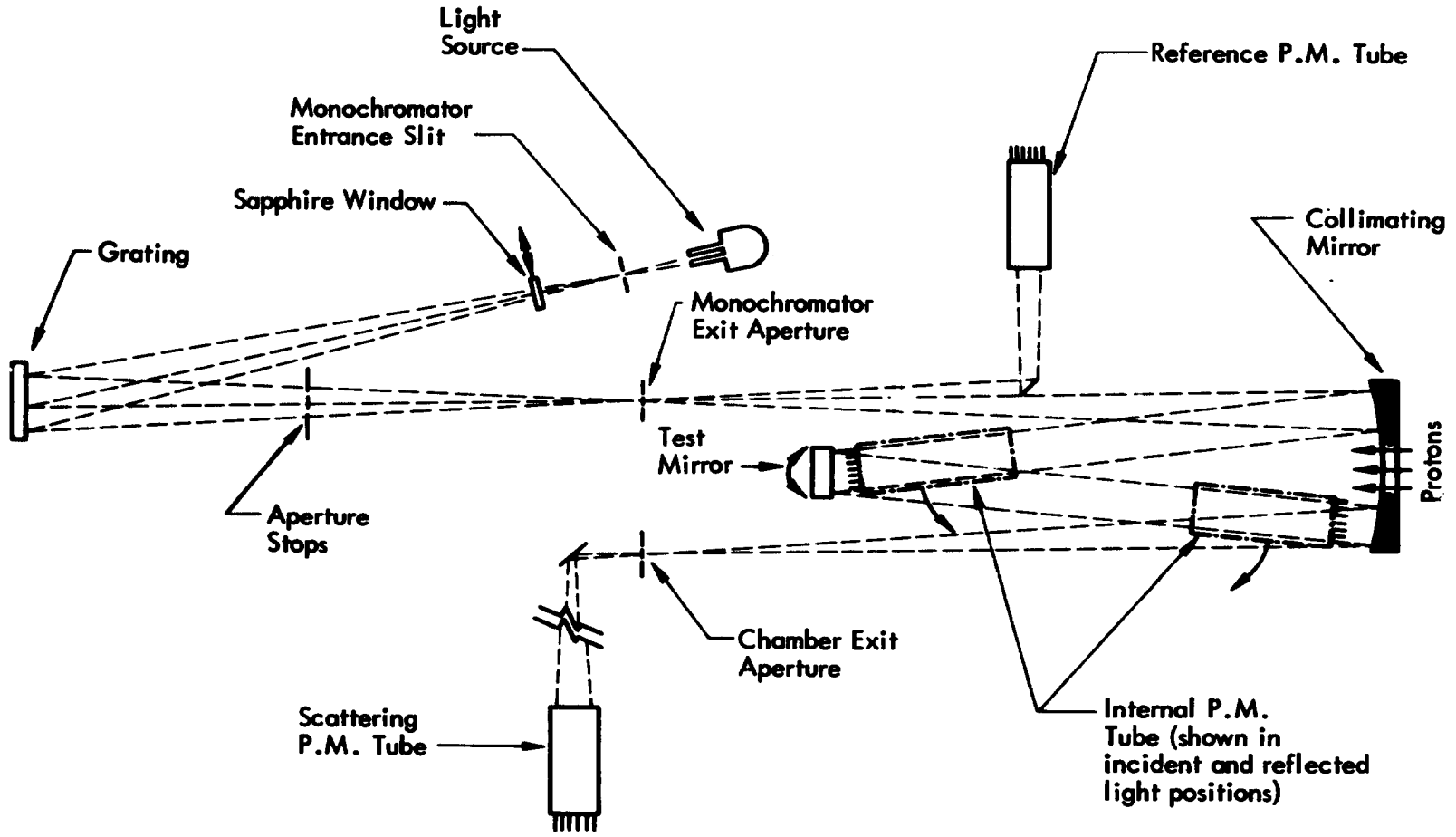


Figure 6: SCHEMATIC OF IN-SITU OPTICAL MEASUREMENT SYSTEM USED FOR MEASURING SPECULAR REFLECTANCE IN THE FAR-ULTRAVIOLET WAVELENGTH REGION, AND SCATTERED LIGHT MEASUREMENTS

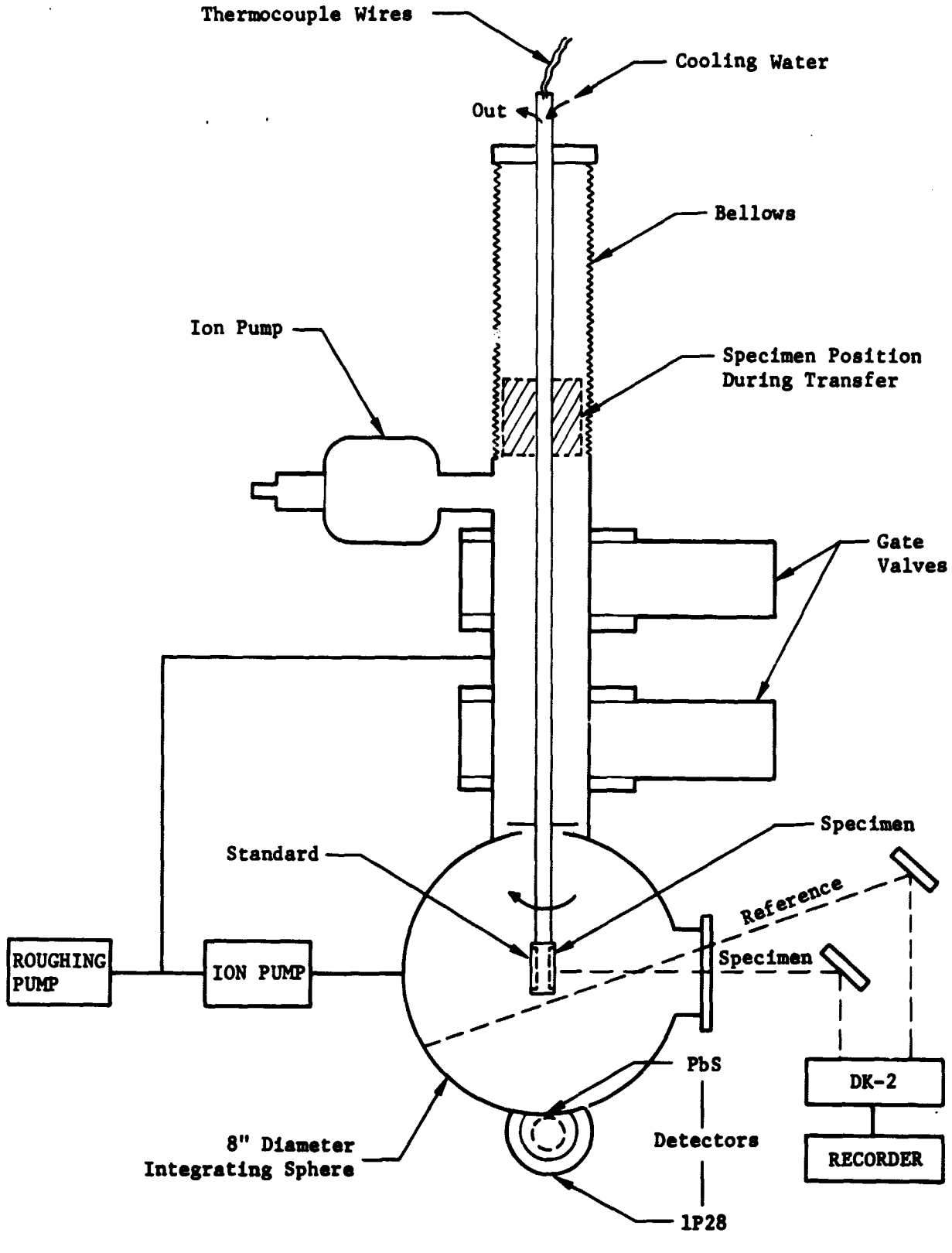


Figure 7. SCHEMATIC OF REFLECTANCE MEASUREMENT FACILITY

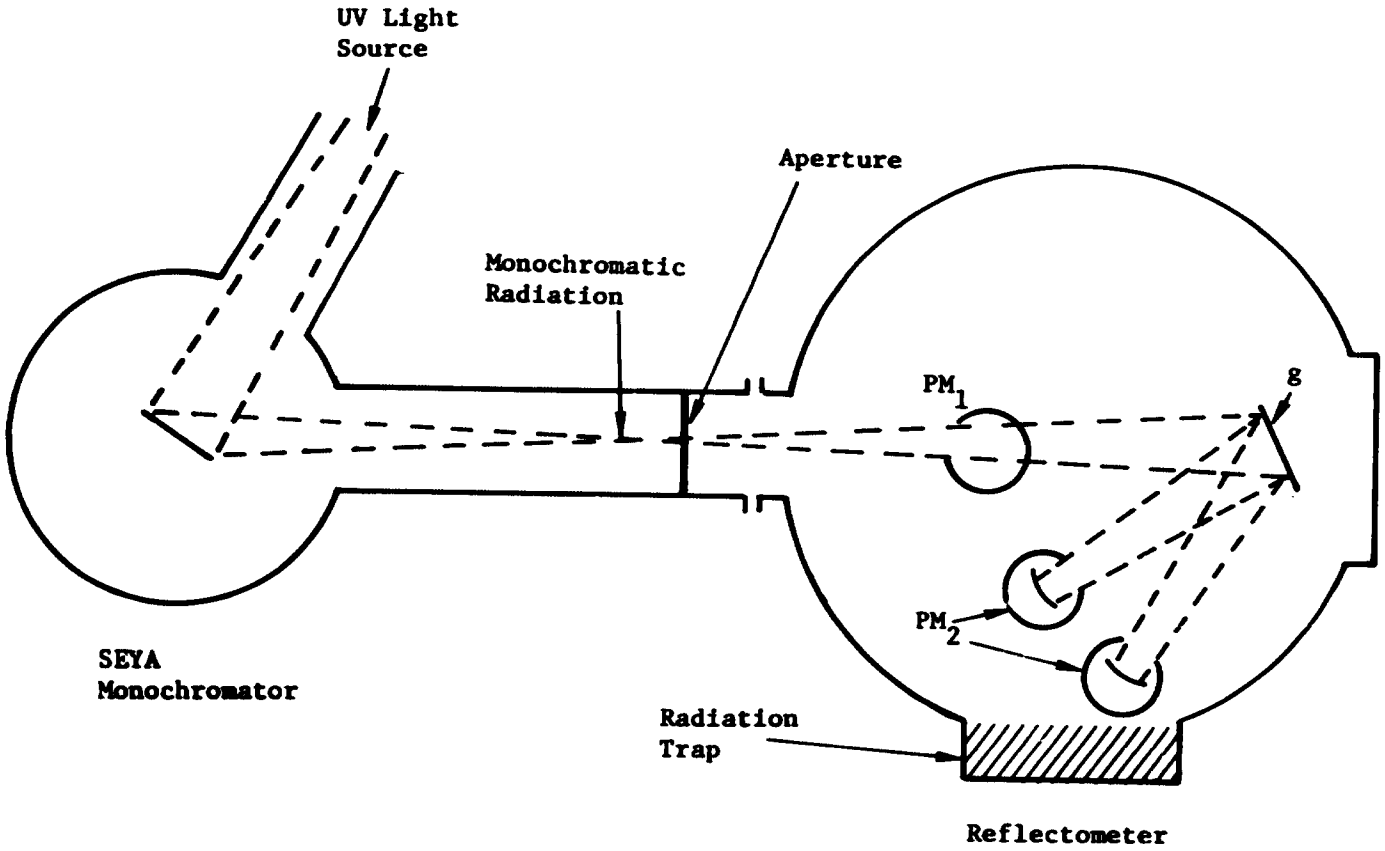


Figure 8: SCHEMATIC OF GRATING EFFICIENCY MEASUREMENT APPARATUS

from the grating is detected by the same photomultiplier in position PM_2. Use of the same detector for both readings minimizes calibration problems. A 5.08 cm diameter, end-window photomultiplier is being used.

6.0 EXPERIMENTAL RESULTS

6.1 Plasma Generator Design Studies

Studies were conducted to determine the optimum method of producing a reactive gas plasma and injecting it into the test chamber. The variables involved are gas flow, vacuum pumping speed, rf power, electrode configuration, plasma inlet configuration, and pressures in the plasma generator and test chamber. Basically, it is desirable to maintain a pressure upwards from 0.5 torr in the discharge region of the plasma generator and less than 10^{-3} torr in the test chamber, utilizing a reasonable size vacuum pump. Two different configurations of the plasma generator were considered: (1) a 15.2 cm diameter glass tube which projected through the wall of the test chamber (Figure 4), and employed a multicapillary end plate for restricting flow; and (2) a series of small diameter quartz tubes which project into the test chamber. In the latter case, flow is restricted by the length and diameter of the quartz tubes.

The design ultimately chosen involves plasma generation in small diameter quartz tubes. The primary reasons for selecting this approach were: (1) recombination of atomic oxygen on tube walls is minimized by high velocity flow; and (2) it provides more flexibility for design changes, i.e., the size, length, and number of tubes used can be easily changed. A series of experiments were conducted in which the pressure drop across quartz tubes of 2, 4, and 5 mm ID was measured for various gas flows. Based on this data a quartz tube of 4 mm ID and about 92 cm length was selected for use on the plasma test facility.

Experiments were then undertaken on the 4 mm ID tube to determine the optimum flow (or upstream pressure) for maximizing atomic oxygen

delivery to the test chamber. The effect of gas flow and rf power was monitored by placing a stainless steel disk directly in front of the exit of a quartz tube (Figure 9), and recording the change in temperature of the disc. It was assumed that temperature would give an indication of the amount of atomic oxygen recombining on the plate, and thus indicate the arrival rate of atomic oxygen. Figure 10 is a plot of the probe temperature versus the upstream pressure for two different rf power settings. The data for 50 watts of power indicates that the maximum arrival rate of atomic oxygen occurs in the range of 40 to 100 torr upstream pressure. Unfortunately, the data does not show the condition at which the maximum percentage of atomic oxygen exists. The optimum operation point for producing the highest percentage dissociation may be at a lower flow. Operating at the latter condition would minimize both pumping problems during tests, and oxygen storage volume in space. It should be noted that the data shown in Figure 10 are subject to convection and conduction cooling effects which vary with pressure. These effects could easily change the shape of the curve and should be accounted for. In regard to rf-power effects, the data shows that additional atomic oxygen can be produced by increasing power from 30 to 50 watts, except in the pressure region below about 5 torr.

It is recognized that much more work needs to be done to determine the optimum conditions for generating plasmas in small quartz tubes; however, these studies are beyond the scope of the present program. A need exists for a comprehensive theory to be developed for predicting the arrival rate of atomic oxygen in a reactive gas plume in high vacuum.

6.2 Liquid Contaminant Inlet System

Two contaminants, urine and ethylene glycol, will be in the liquid state when applied to the test specimens. The other two contaminants, styrene and methyl trimethoxysilane, can be evaporated and bled into the chamber as gases.

Experiments were conducted to develop a satisfactory method for injecting liquids into the vacuum so that a uniform coating can be applied to test specimens. Numerous approaches for injecting liquids were tryed. The approach selected utilizes a modified oil burner nozzle as shown in Figure 11. Compressed gas is used to force the liquid through the nozzle.

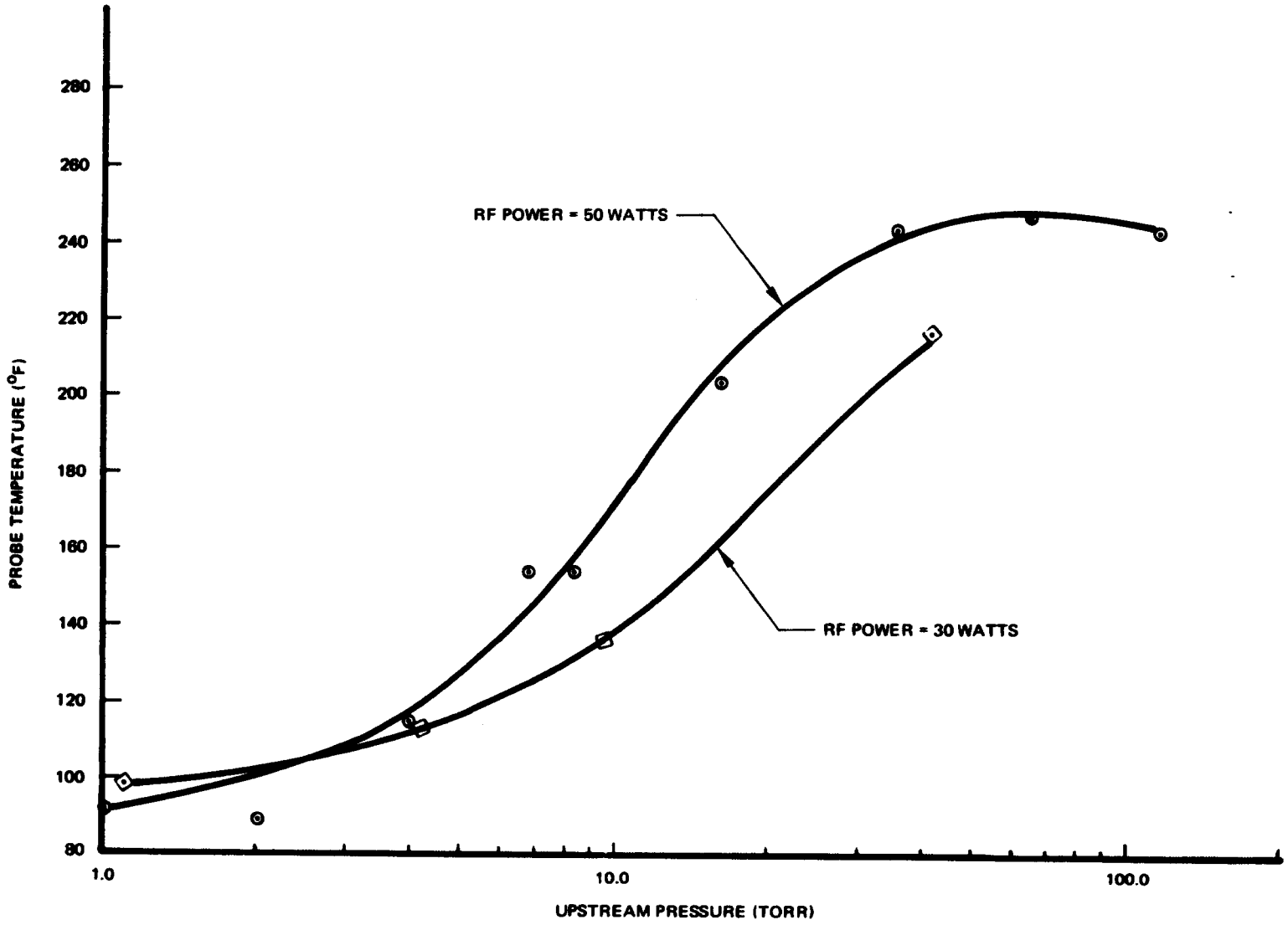


Figure 10: STAINLESS STEEL PROBE TEMPERATURE VS UPSTREAM PRESSURE FOR 4 MM I.D. QUARTZ TUBE

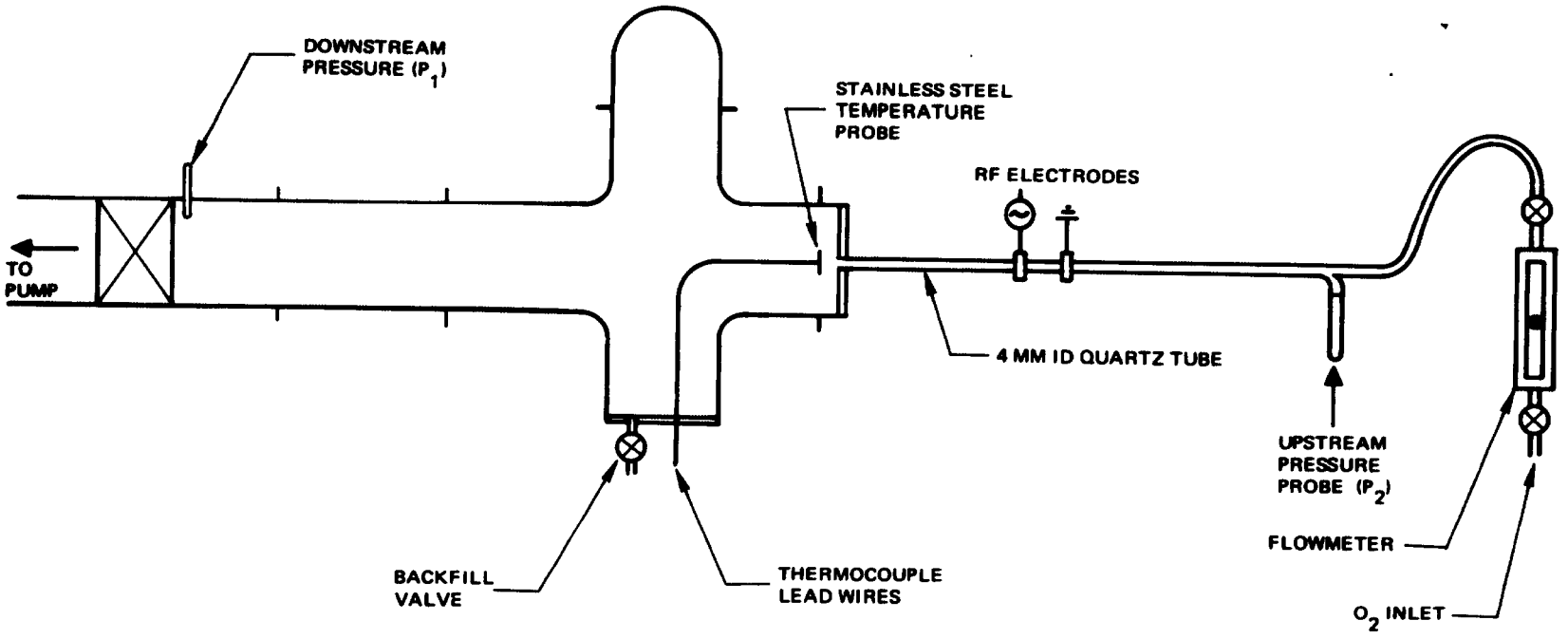


Figure 9: OXYGEN PLASMA TEST SYSTEM WITH SMALL DIAMETER QUARTZ TUBE

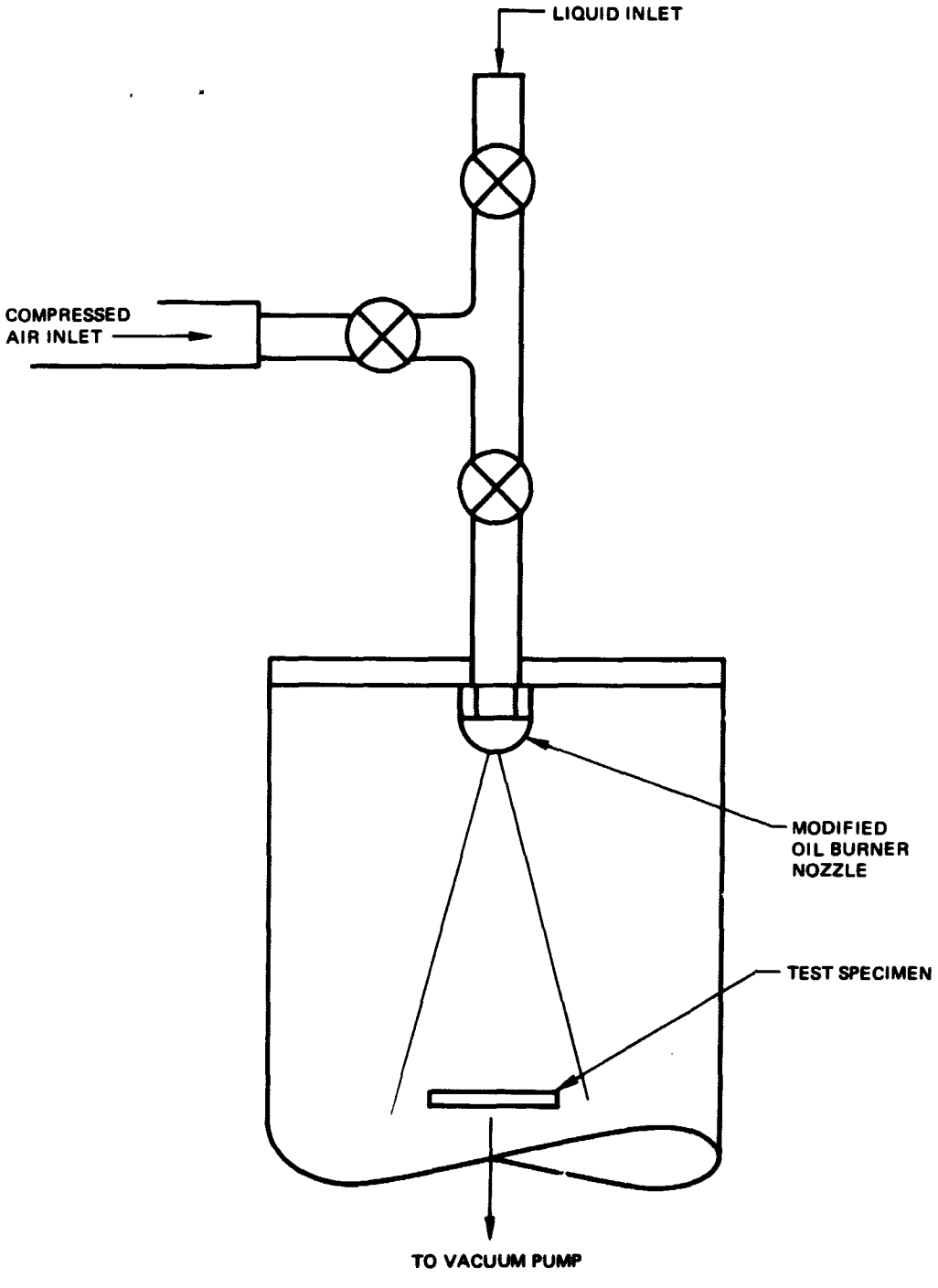


Figure 11: LIQUID CONTAMINANT INLET SYSTEM

7.0 PROGRAM PROGRESS

7.1 Schedule

A schedule for the overall program is shown in Figure 12. Solid bars represent work completed, and open bars work planned. It is planned to complete all work in twelve months. The first three months were devoted to preparation of facilities and test specimens. Exploratory experiments are planned during the fourth and fifth months, and comprehensive testing will be accomplished in the sixth to ninth month. If a supplemental cleaning technique needs to be evaluated on certain specimens, a one month period is reserved for this work in the ninth and tenth month.

Design studies will be conducted during the tenth month. A draft of the final report will be submitted at the end of the eleventh month. Release of the final report is planned by the end of the twelfth month.

An analysis of work completed at the end of the first quarter showed that preparation of test specimens and test facilities will probably continue for several weeks. Thermal control surfaces have been received from NASA, and the gold and platinum coated mirrors are complete. Preparation of facilities is approximately 90 percent complete.

7.2 Expenditures

Cumulative expenditures as of May 1, 1971 were about $7462. Approximately $37,111 will be required to complete the program, including fee. No cost overrun is anticipated.

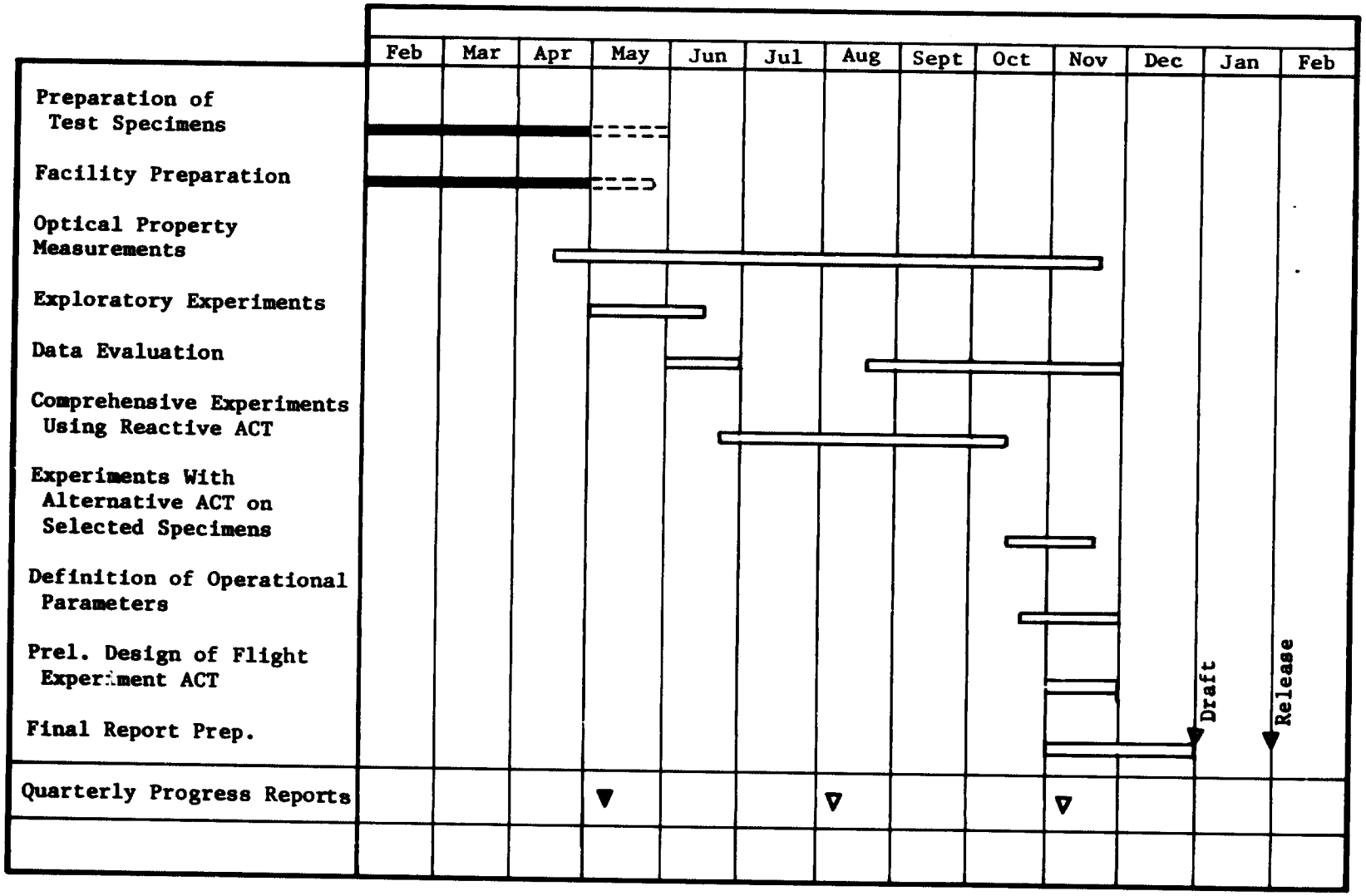


Figure 12: PROGRAM SCHEDULE

7.3 Future Work Planned

During the next reporting period the following items will be initiated or completed:

1. Preparation of test specimens and facilities will be completed.
2. Exploratory experiments will be completed.
3. Comprehensive experiments will be initiated.

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